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# THE SYNERGY OF THE SURFACE LAYER AFTER CARBURIZING OF TOOL STEEL AS A MEASURE OF OPERATIONAL QUALITY

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In the paper the results of the investigation on surface layer of carburized X150CrMoV12-1 tool steel are presented. Abrasive wearing of the surface was determined according the ASTM G77-98 method. The microstructure of the matrix and distribution of carbides were observed in a Scanning Electron Microscope (SEM). A correlation between the investigated parameters (microstructure/carbides distribution) was found.

***Key words:*** *tool steel, surface layer, carburization, microstructure, friction*

**Sinergija površinskog sloja alatnog čelika poslije naugljičenja kao pokazatelj uporabne kakvoće.** Članak daje rezultate istraživanja naugljičenog površinskog sloja alatnog čelika X150CrMoV12-1. Abrazivno trošenje površine određeno je sukladno metodi ASTM G77-98. Mikrostruktura materijala i raspodjela karbida promatrana je na skening elektronskom mikroskopu (SEM). Utvrđena je zavisnost između istraživanih parametara (raspodjela mikrostruktura/karbidi).

***Ključne riječi:*** *alatni čelik, površinski sloj, naugljičenje, mikrostruktura, trenje*

## INTRODUCTION

The operational durability of the surface layer of tool steels continues to be of basic importance for process and design engineers. Among numerous types of wear met, abrasive or adhesive wear in a basic type [1, 2]. Abrasive action occurrence, when hard particles produced by the wear of friction pair, or deriving from the environment, appear in the friction nodes. By contrast, in the case of adhesive wear, particles torn out from one or both friction pairs bind permanently to one of the friction surfaces [3 - 6]. Investigations have been carried out for a number of years with the aim of optimizing the properties of the operational surface of tools. However, the surface layer, as a heterophase system, should be considered in a detail sufficiently great to find definitely the best (for given operation conditions) interrelationship between phases occurring in the surface layer.

In technology, synergism occurs at all stages of the manufacture and operation of products, starting from the acquisition of raw-materials to finished products [7].

The synergistic factors can be classified in technology into the following types: parametrical (selection of manufacturing parameters), material (chemical elements and their compounds), technological (compositions of aerologic systems manufacture and operation technologies) and constructional (compositions of different constructional elements) [8, 9].

From among many technologies forming the aerologic system, which is the surface layer, the best understood and the most commonly used technology is carburization. Until recently, this process was applied for low-carbon steels; however, also medium- and high-carbon steels are increasingly often submitted to carburizing. During the process of carburizing high-carbon steels, dispersion carbides form and grow as early as at the stage of diffusion, with amount and shape undergoing some changes during subsequent tempering. The carbide phase is not a homogeneous phase, either in terms of chemical composition, shape, or size; nevertheless, when interacting with the metallic matrix, of tempered martensite, this phase forms a synergic system, whose synergy effect is defined as the quality of the aerologic system.

## TESTING RESULTS

Ledeburitic cold-work tool steel, grade X150CrMoV12-1, was selected for the testing. This steel was designed for operation under the conditions of dry friction.

The great popularity, enjoyed, has made it one of the basic ledeburitic steels for tests aimed to modify the surface layer. The chemical composition of the steel is given in Table 1.

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Table 1. **Chemical Compositionof the X150CrMoV12-1 steel wt %**
Tablica 1. **Kemijski sastav čelika X150CrMoV12-1, tež. %**

| C | Mn | Si | Ni | Cr |
|---|---|---|---|---|
| 1,65 | 0,299 | 0,153 | 0,235 | 11,58 |
| Mo | V | S | P | Cu |
| 0,662 | 1,03 | 0,0154 | 0,02 | 0,0987 |

Specimens prepared of this steel had in the initial state two different structures; concerning both, the degree of refinement of ledeburitic carbides and the degree of dispersion and spheroidization of dispersion carbides. The microstructure of the steels tested are shown in Figure 1.

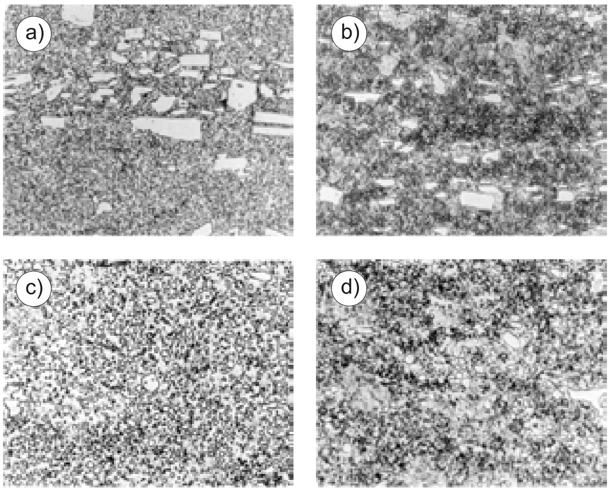



Figure 1. **Microstructure of X150CrMoV12-1 steel in as received state, etch. nital, a) steel 1 magn. 500×, b) steel 2 magn. 500×, c) steel 1 magn. 1000×, d) steel 2 magn. 1000×**
Slika 1. **Mikrostruktura čelika X150CrMoV12-1 u polaznom stanju, jetkano nitalom; a) čelik 1, povećanje 500 ×, b) čelik 2, povećanje 500 ×, c) čelik 1, povećanje 1000 ×, d) čelik 2, povećanje 1000 ×**

The thermochemical treatment included the following stages:

- heating: $T$ = 823 K, $\tau$ = 1,8 ks,
- austenitizing and carburizing: $T$ = 1273K, $\tau$ = 3,6 ks, $\alpha_p$=0,22, and
- tempering: $T_1$ = 723 K, $T_2$ = 823 K, $T_3$ = 823 K, $\tau_{1,2,3}$ = 7,2 ks.

The surface layers, obtained as a result of carburization, are shown in Figure 2.

Hardness is the resultant feature of the dispersion and size of carbides and the degree of matrix supersaturation.

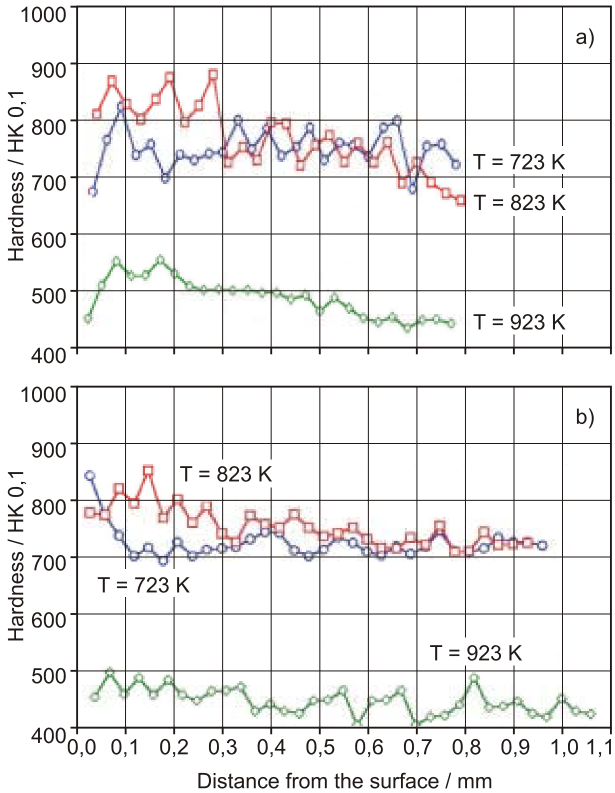



Figure 3. **Distribution of microhardness in surface layer of X150CrMoV12-1 steel after carburizing at 1273K/3,6 ks and tempering for 7,2 ks at the temperatures of 723 K, 823 K, 923 K; a) steel 1, b) steel 2**
Slika 3. **Raspodjela mikrotvrdoće na površinskom sloju čelika X150CrMoV12-1 poslije naugljičenja na 1273K/3,6 ks i temperinga za 7,2 ks na temperaturama: 723 K, 823 K, 923 K; a) čelik 1, b) čelik 2**

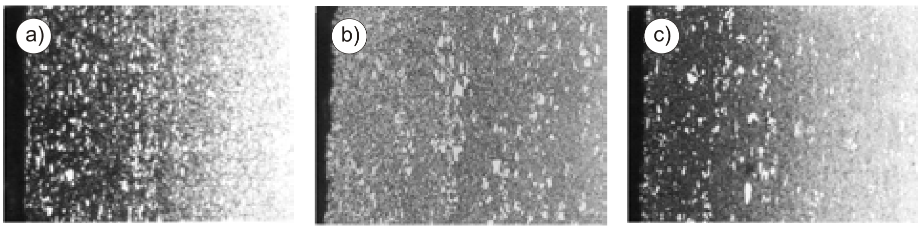



Figure 2. **Microstructure of the surface layer of steel X150CrMoV12-1 after carburizing at 1273 K/3,6 ks and tempering for 7,2 ks and the temperatures of a) 723 K, b) 823 K, c) 923 K, etch. nital, magn. 200×**
Slika 2. **Mikrostruktura površinskog sloja čelika X150CrMoV12-1 poslije naugljičenja na 1273 K/3,6 ks i temperinga za 7,2 ks na temperaturama: a) 723 K, b) 823 K, c) 923 K jetkano nitalom, povećanje 200 ×**

However, due to the structural inhomogeneity of the steels (the presence of large ledeburitic carbides), the obtained surface layers were tested for microhardness. The microhardness tests were performed by the Knoop method with a load of 0.980 N on a semi-automatic microhardness tester, type FM-7, supplied by Future Technology Corp. The results are shown in Figure 3.

The microhardness is a resultant of the size, dispersion and shape of carbides and the degree of matrix over saturation. For the comprehensive determination of the interrelation between the carbide phase and the metallic matrix, both phases were submitted to individual tests. The testing of carbides particle, was aimed at establishing their kind (chemical composition and type) and determining their size and shape.

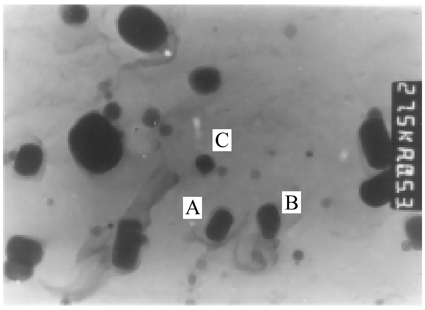
The qualitative estimation of carbides was adapted EDX analysis and electron diffraction. The results of there tests the carburized steel in Figure 4. show that the carbides contain primarily of Cr, Fe and Mo.

The results of the electron diffraction-based qualitative tests on are presented in Figure 5.

The performed analysis of extraction carbon replicas allowed the identification of the locations of occurrence and the morphology of dispersion carbide particles precipitated during the tempering. The results of the examination are shown in Figures 6. - 8.

During the thermal and thermochemical treatments, dispersion carbide particles undergo changes in size and shape. In the initial state the steel taken for testing differed in the extent of carbides dispersion. In order to determine the effect of thermochemical treatment on the change of size of the dispersion carbide particles, their plane section area and shape factor were measured. To describe accurately the change of size of carbide particles, the measurements were taken in two ranges of plane section area: $A_A \geq 0.05$ $\mu m^2$ and $A_A < 0.05$ $\mu m^2$. The results are given in Table 2.

The shape factor, $\eta A_A$, for carbide particles examined was defined as percentage of similarity to the shape




| Carbide, A | wt. / % | at. / % |
|---|---|---|
| V | 2,48 | 2,66 |
| Cr | 51,12 | 53,67 |
| Fe | 42,27 | 41,32 |
| Mo | 4,11 | 2,34 |

| Carbide, B | wt. / % | at. / % |
|---|---|---|
| V | 2,8 | 3,02 |
| Cr | 50,4 | 53,17 |
| Fe | 41,4 | 40,78 |
| Mo | 5,4 | 3,03 |

| Carbide, C | wt. / % | at. / % |
|---|---|---|
| V | 16,25 | 17,92 |
| Cr | 43,35 | 45,27 |
| Fe | 34,5 | 33,33 |
| Mo | 5,9 | 3,3 |

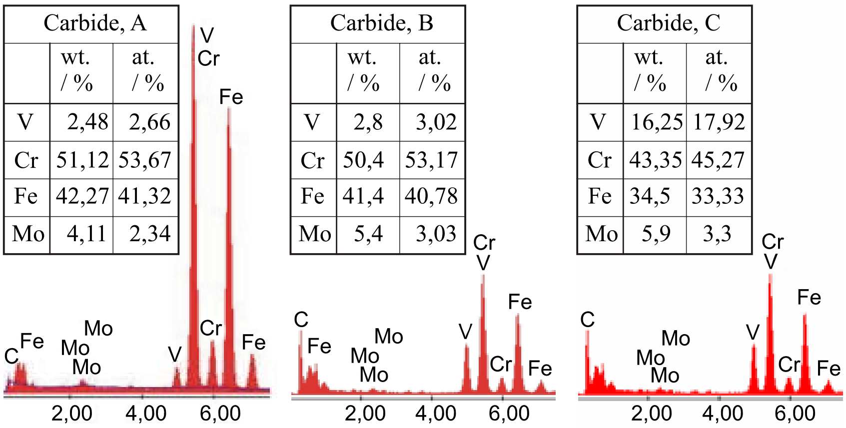



Figure 4. **Results of the analysis of chemical composition of carbide particles in the surface layer of steel X150CrMoV12-1 after carburizing at 1273K/3,6 ks**

Slika 4. **Rezultati analize kemijskog sastava čestica karbida na površinskom sloju čelika X150CrMoV12-1 poslije naugljičenja na 1273K/3,6 ks**

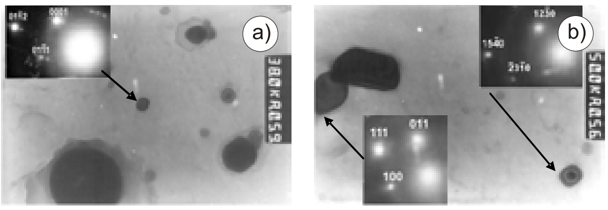



Figure 5. **Microstructure and diffraction patterns of carbide particles in the surface layer of steel X150CrMoV12-1 after carburizing at 1273K/3,6 ks (carbides of types $M_3C$ and $M_7C_3$**

Slika 5. **Mikrostruktura i difrakcijski prikaz čestica karbida na površinskom sloju čelika X150CrMoV12-1 poslije naugljičenja na 1273K/3,6 ks (karbidi vrste $M_3C$ i $M_7C_3$)**

Table 2. **Results of the measurements of the average plane section area**

Tablica 2. **Rezultati mjerenja prosječnog djela površine**

| | $A_A$, Steel 1 | | $A_A$, Steel 2 | |
|---|---|---|---|---|
| | ≥0,05 $\mu m^2$ | <0,05 $\mu m^2$ | ≥0,05 $\mu m^2$ | <0,05 $\mu m^2$ |
| after carburizing | 0,71 | 0,025 | 0,21 | 0,0248 |
| after tempering | | | | |
| 723K | | 0,00136 | | 0,0046 |
| 823K | | 0,0028 | | 0,0061 |
| 923K | | 0,0041 | | 0,0078 |

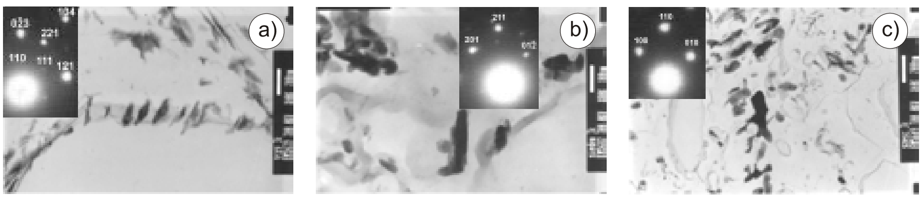



Figure 6. **Microstructure and diffraction patterns of $M_3C$-type carbide particles in the surface layer of steel X150CrMoV12-1 after carburizing at 1273K/3,6 ks and tempering for 7,2 ks and the temperatures of a) 723 K, b) 823 K, c ) 923 K, extraction replica**

Slika 6. **Mikrostruktura i difrakcijski prikaz $M_3C$ vrste karbidnih čestica na površinskom sloju čelika X150CrMoV12-1 poslije naugljičenja na 1273K/3,6 ks i temperinga za 7,2 ks na temperaturama: a) 723 K, b) 823 K, c ) 923 K (primjena replike)**

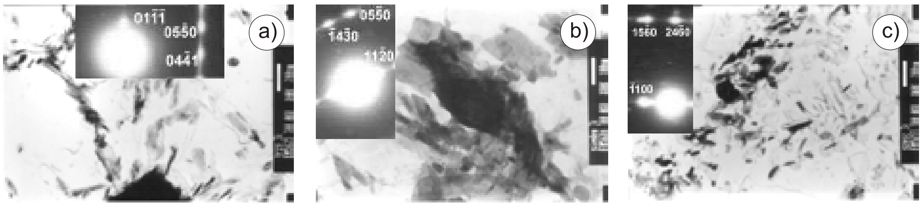



Figure 7. **Microstructure and diffraction patterns of $M_7C_3$-type carbide particles in the surface layer of steel X150CrMoV12-1 after carburizing at 1273K/3,6 ks and tempering for a 7,2 ks and the temperatures of a) 723 K, b) 823 K, c) 923 K, extraction replica**

Slika 7. **Mikrostruktura i difrakcijski prikaz $M_7C_3$ vrste karbidnih čestica na površinskom sloju čelika X150CrMoV12-1 poslije naugljičenja na 1273K/3,6 ks i temperinga za 7,2 ks na temperaturama: a) 723 K, b) 823 K, c) 923 K (primjena replike)**

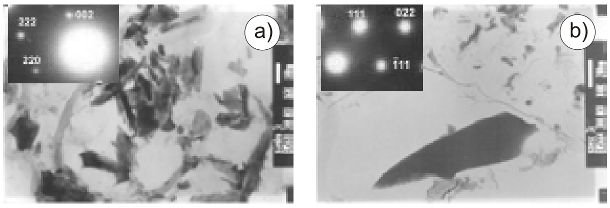



Figure 8. **Microstructure and diffraction patterns of $M_{23}C_6$-type carbide particles in the surface layer of steel X150CrMoV12-1 after carburizing at 1273K/3,6 ks and tempering for 7,2 ks and the temperatures of a) 823 K, b) 923 K, extraction replica**

Slika 8. **Mikrostruktura i difrakcijski prikaz $M_{23}C_6$ vrste karbidnih čestica na površinskom sloju čelika X150CrMoV12-1 poslije naugljičenja na 1273K/3,6 ks i temperinga za 7,2 ks na temperaturama: a) 823 K, b) 923 K (primjena replike)**

of a circle. The results of shape factor examination are shown in Table 3.

Table 3. **The results of shape factor measurements**

Tablica 3. **Rezultati mjerenja frakture oblika**

| | $\eta\ A_A$, Steel 1 | | $\eta\ A_A$, Steel 2 | |
|---|---|---|---|---|
| | ≥0,05 % | <0,05 % | ≥0,05 % | <0,05 % |
| after carburizing | 55,9 | 81,59 | 65,49 | 80,60 |
| after tempering | | | | |
| 723K | | 49,29 | | 45,80 |
| 823K | | 40,51 | | 54,46 |
| 923K | | 50,10 | | 63,75 |

The change in the chemical composition and type of carbide particles is due to of both the conditions of thermochemical treatment and chemical composition of the over saturated matrix. In order to determine the changes in chemical composition of the matrix resulting from the saturation of austenite with carbon, as well as from the decomposition of martensite during tempering, X-ray examination was performed. The variation of the carbon content of austenite, as a function of the distance from the surface, is shown in Figure 9.

The tempering of the previously carburized tool steel, in addition to the precipitation and growth of the dispersion carbide phase, causes the transformation of martensite and change of it tetragonal structure. The results of examination of the change in the lattice constant parameter and the tetragonal structure of martensite as a function of tempering temperature are given in Table 4.

Table 4. **The results of measurements of the lattice constant and tetragonality of martenzite**

Tablica 4. **Rezultati mjerenja konstante rešetke i tetragonalnosti martenzita**

| | Steel 1 | | Steel 2 | |
|---|---|---|---|---|
| | lattice constant / nm | $a_c - a_{a,b}$ / nm | lattice constant / nm | $a_c - a_{a,b}$ / nm |
| 723K | 2,872 | 0,019 | 2,878 | 0,012 |
| 823K | 2,869 | 0,015 | 2,871 | 0,010 |
| 923K | 2,868 | 0,012 | 2,869 | 0,006 |

The X150CrMoV12-1-grade tool steel tested is a steel designed for cold work. The performed surface treatment was intended to increase substantially it abrasive wear resistance, increasing thereby the quality of manufactured tools.

In order to verify the quality of the performed treatment, technological tests were carried out. The tribological tests were carried out according to the ASTM G77 standard, on

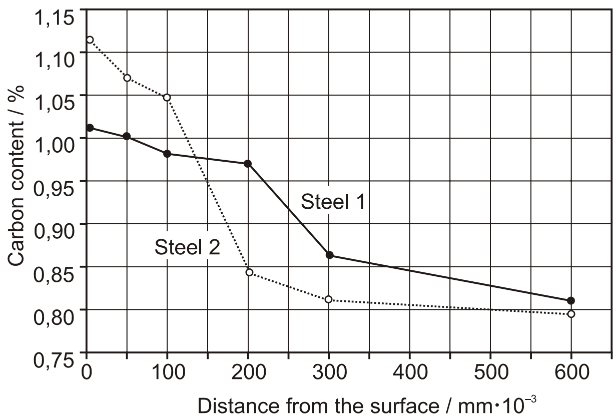



Figure 9. **Variation of carbon content in the matrix of the top layer of steel X150CrMoV12-1 after carburizing at a temperature of 1273 K for 3,6 ks**

Slika 9. **Promjene sadržaja ugljika u matriksu na vrhu sloja čelika X150CrMoV12-1 poslije naugljičenja na temperaturi 1273 K za 3,6 ks** specimens after thermal and thermochemical treatments. The testing results are shown in Figure 10.

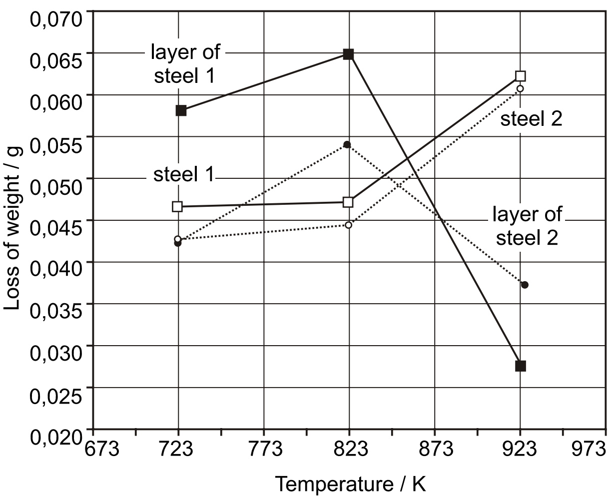



Figure 10. **Results of the testing of steel X150CrMoV12-1 linear friction velocity, *v* = 1 m/s; loading, *P* = 53 N; friction time *t* = 15 ks**

Slika 10. **Rezultati ispitivanja čelika X150CrMoV12-1 linearna brzina trenja, *v* = 1 m/s; opterećenje, *P* = 53 N; vrijeme trenja *t* = 15 ks**

With a view to obtaining additional information on the mechanism of wear during abrasive action, the examination of the surfaces of X150CrMoV12-1 steel specimens were performed on a JEOL JSM 5400 scanning microscope (Figure 11.).

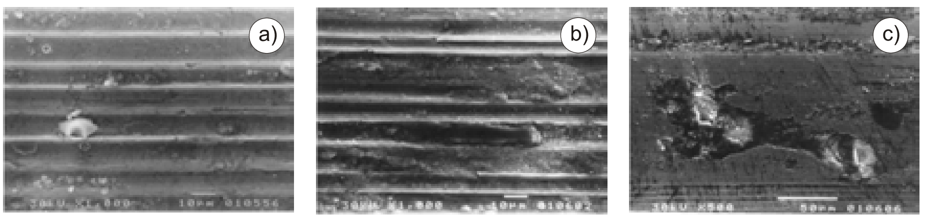



Figure 11. **Effects of the wear of specimens upon the abrasion of the X150CrMoV12-1 tested after carburizing (1273K/3,6 ks) and tempering: a) 723K/7,2 ks, b) 823K/7,2 ks c) 973K/7,2 ks**

Slika 11. **Efekt trenja uzoraka uslijed abrazije X150CrMoV12-1 ispitivanog poslije naugljičenja (1273K/3,6 ks) i temperinga: a) 723K/7,2 ks, b) 823K/7,2 ks c) 973K/7,2 ks**

## ANALYSIS OF THE TESTING RESULTS

The carburizing of the X150CrMoV12-1 high-alloy tool steel introduces substantial changes to the surface layer as a result of changes of the carbide phase and the matrix. The carbide phase is responsible for the properties of the surface layer. The tests carried out have shown that after carburizing the main carbides present in the structure are of $M_3C$ and $M_7C_3$ types, with Cr, Fe, V and Mo in their composition. The performed tempering causes carbides of $M_3C$, $M_7C_3$, $M_{23}C_6$ types to precipitate from the oversaturated solution, in martensite, with the carbides of $M_{23}C_6$ type occurring only after tempering at a temperature above 823 K. The stereological examination showed that the average plane section of dispersion carbides increased with increasing tempering temperature, while clearly larger carbides were observed in steel 2. The distinct effect of tempering temperature on the spheroidization of carbides was confirmed by shape factor examination; in the case of steel 2, a higher degree of spheroidization was observed (after tempering at 923 K it amounted to 63,75 %).

The matrix in successive stages of thermochemical treatment is a complex of austenite, martensite and tempered martensite. The measurements of carbon content in the surface layer, as dependent on the initial microstructure, showed a higher carbon concentration in the matrix of a specimen of steel with a lower degree of carbide phase dispersion (1,11 % C); whereas, for a specimen of steel with a lower degree of carbide phase dispersion, the carbon concentration on the surface amounted to 1,01 % (Figure 9.). The completed tempering of the carburized X150CrMoV12-1 did not only cause the precipitation of carbides, but also the transformation of the matrix. The measurements of the lattice constant of martensite and its tetragonality, as given in Table 4., showed that the increase in tempering temperature caused a clear decrease of the lattice constant and a lowering of the degree of martensite tetragonality.

The microhardness tests carried out (Figure 3.) and abrasion resistance tests made it possible to determine the influence of the performed fluidized-bed thermochemical treatment of steel X150CrMoV12-1 on the service properties as the result of the synergic effect of the carbide phase and the metallic matrix.

The patterns of friction wear of specimens tempered at temperatures of 723 K and 823 K are very similar (Figures 11.a, b). In both cases, parallel grooves has formed over the whole abrasion width. At the groove bottoms of the specimen tempered at 823 K, crushing of abraded material products occurs, while at the groove bottoms of the specimen tempered at 723 K, spalling of the material is visible. The surface of the top layer of the X150CrMoV12-1 steel specimen after tempering at 923 K is shown in Figure 11.c. The visible differences in the abrasive wear mechanism, as compared to the specimens of steel tempered at temperatures 723 K and 823 K, are linked with both the size and shape of carbide particles and the low hardness of the matrix.

The abrasive wear of the X150CrMoV12-1 steel specimen tempered at 923 K occurs only to a negligible extent as a result of cutting-grooving (Figure 11.c) - the main cause of the abrasive wear is spalling.

## CONCLUSIONS

1. Carburizing of the X150CrMoV12-1 high-alloy steel results in the formation of a heterophase system in the surface layer, in which interrelations between phases determine the operational properties of the surface layer.
2. The abrasion of X150CrMoV12-1 steel specimens after tempering at 723 K and 823 K is accompanied by an abrasive wear mechanism caused by carbide particles being torn out from the high-hardness matrix.
3. The increase in the wear resistance of the carburized X150CrMoV12-1 steel surface layer is a result of the greater relative volume of the spheroid-shaped carbide phase located in the low-hardness matrix, which causes carbide particles to be pressed into the high-tempered martensite matrix during abrasion, causing the change in the wear mechanism from abrasive to fatigue type.

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